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REMARKS

Examiner L. Umez-Eronini is thanked for the thorough examination and search of the subject Patent Application. Claims 3, 4, 8, and 9 have been amended.

The Specification and claims 3, 4, 8, and 9 have been amended to overcome rejection under 35 U.S.C. 112, second paragraph. The trademarks/trade names have been defined by their chemical makeup, according to Internet sources found. Please see the exhibits for definitions of the materials as follows: HOSP (exhibit A), SILK (exhibit B), FLARE (exhibit C), CORAL (exhibit D), Black Diamond (Exhibit E), Z3MS (exhibit F), XLK (exhibit G).

The Examiner is thanked for allowing Claims 1, 2, 5, 6, 7, and 10.

All Claims are believed to be in condition for Allowance, and that is so requested.

It is requested that should Examiner Umez-Eronini not find that the Claims are now Allowable that she call the undersigned at 765 4530866 to overcome any problems preventing allowance.

Respectfully submitted,



Rosemary L. S. Pike. Reg # 39,332

Attachment: Exhibits A-G

Exhibit A

**looksmart**

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☐ Advanced Search ☐YOU ARE HERE: [Articles](#) > [Electronic News](#) > [Dec 7, 1998](#) > Article[Print article](#) [Tell a friend](#) [Find subscription deals](#)**AlliedSignal Unveils HOSP.(Technology Information)(Column)****Electronic News**, Dec 7, 1998

tokyo -- AlliedSignal unveiled the third platform of its low-k dielectric materials program, a hybrid of its Flare, organic materials-based spin-on dielectric and Nanoglass, an inorganic product family.

"Not just a mixture, but a hybrid material that is essentially SiO<sub>2</sub>-based," according to an AlliedSignal spokesperson, the hybrid material dubbed HOSP (Hydrido Organo Siloxane Polymer) currently carries a k constant of 2.5 and is suitable for sub 0.18-micron processing. The material can be scaled down beyond the k equals 2 constant threshold as well. AlliedSignal plans to scale each of its three platforms down below this threshold.

In a conference call, AlliedSignal boasted that HOSP can be integrated into both subtractive aluminum and damascene processes. The company claims HOSP provides low stress, thermal stability to 550 degrees, and 1-micron single coat crack-free films.

The material has been evaluated with no liner, but has had a cap put on. "We do not know if the cap is necessary yet," said Jack Bollick, VP/GM for AlliedSignal Electronic Materials' Wafer Fabrication group.

HOSP is being produced at an AlliedSignal site in the Midwest. Mr. Bollick declined to specify where due to competitive reasons.

Within the next 12 months, AlliedSignal expects HOSP as well as its other low-k dielectric lines to all be integrated in pilot lines.

Asked about his assessment of Applied Materials, Black Diamond low-k chemical vapor deposition (CVD) film, Bollick said, "I think like all the other competitors it is a very formidable," however, "AlliedSignal is betting a lot of money on it, we feel we have the right tools and the right technology to compete in the arena."

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## What's Up From SEMI - Industry Report

**August 2001**

### *Innovations in Interconnect Technology*

*by Neil H. Hendricks, Ph.D, Chief Technologist, Material Lifecycle Solutions, ATMI, and co-chairman of the SEMI Technical Symposium (STS) session on "Innovations in Interconnect Technology" that was held on July 16, 2001 in San Francisco, California. .*

The Innovations in Interconnect Technology technical session (STS) at SEMICON West 2001 featured several timely and important discussions focused on implementation of copper/low k damascene at both the 0.13m and 0.10m technology nodes. Many issues related to materials selection, scaling, and process integration were addressed by leading industry experts. The session was intentionally configured to include leading edge contributions from chip manufacturers, OEM equipment manufacturers, and semiconductor material suppliers.

Dr. Bradley Melnick from the Motorola Semiconductor Products Sector began the session with a discussion of scaling challenges in interconnect technology for 0.13m and beyond. As one of the earliest adopters of copper wiring, Motorola's perspectives on the scaling challenges were of great interest to the Technology Symposium attendees. Dr. Melnick addressed the challenges outlined in the technology roadmaps associated with interconnect, dielectrics, and lithography. He also discussed Motorola's "HiPerMOS" technology roadmap, which details Motorola's proposed materials and interconnect architecture through the 0.05 micron generation. Motorola manufactures many types of devices. Many incorporate high performance logic, mixed signal, low power and embedded memory devices. Specific modules have been developed for contact, tight pitch, intermediate pitch and final metal layers. These modules can be combined as needed to build devices with 9 metal levels.

Dr. Melnick showed that each module consisted of patterning, via etch and clean, barrier and copper seed layer deposition, electrodeposition and CMP. Each of these steps had to be completely understood and the conditions optimized in order to meet tight tolerances imposed by the 0.13 micron design rules.

A key, widely discussed challenge for multilevel copper wiring relates to controlling corrosion and related problems; Dr. Hartmut Ruelke from Advanced Micro Device's Saxony Manufacturing facility in Dresden discussed copper surface treatment in damascene technology, with a focus on the strategies and tools used to ensure a reliable interface between copper and adjacent dielectric layers. Dr. Ruelke's talk focused on the use of "reducing plasmas" containing hydrogen and/or ammonia in an AMAT PE-CVD tool to remove copper oxide

prior to dielectric deposition. The greatest improvement in adhesion between copper and the nitride capping layer was achieved with relatively long ammonia plasma treatments using fairly high RF power. Dr. Ruelke showed that electromigration reliability could be significantly improved using the ammonia plasma treatment.

Dr. Michael Mills from Dow Chemical presented on the use of SiLK™ Low K spin-on dielectric (SOD) for intermetal dielectric layers. Noting that IBM and others have announced their intention to use SiLK™ in production at 0.13 microns, Dr. Mills described distinct integration schemes which lead to substantially different effective K values, depending on whether or not buried etch stops, or a hybrid ILD scheme is used. The molecular structures of the precursor materials to SiLK™ were shown, indicating that the final film is a highly crosslinked polyphenylene polymer.

Dr. Mills also described Dow's development of a porous version of SiLK™, designed for ultra-low K applications, for example for 0.10 or 0.07 micron design rules. Data was presented indicating the potential viability of porous SiLK™ as an IMD; for example, metal layers above porous SiLK™ were shown to be removed by CMP using the same polishing process as for non-porous SiLK™.

Dr. Luping Wang from ATMI presented on an innovative, sub-atmospheric chemical delivery system for trimethylsilane (3MS). 3MS is an important chemical for the preparation of Low K films, and is used in processes developed by a major CVD equipment OEM. Dr. Wang described how efficient packaging and transfer of 3MS to multiple CVD platforms is required for high volume manufacturing. Dr. Wang described several benefits result from sub-atmospheric 3MS delivery. Safety, throughput, and cost-of-ownership are favorably impacted by the use of this unique chemical delivery technology.

While the first four presentations in the Innovations in Interconnect session were focused largely on process integration issues at the 0.15-0.13m technology node, the final two presentations of the session were focused on more futuristic, 0.10m and perhaps 0.07m technologies.

Dr. Herman Meynen discussed Dow Corning's development of ultra-low K ( $K < 2.2$ ) spin-on porous dielectric films. He provided a significant update to the initial work described by Dow Corning over a year ago. Working with researchers at IMEC (Leuven, Belgium), Dow Corning has made substantial progress in their work to successfully integrate this ultra-low K material (XLK™). The film, which has a K value of about 2.0, contains 50% porosity. The process described included IMP TaN, then Cu seed sputtering, followed by copper electroplating. Interestingly, Dr. Meynen noted that the metal-filling processes do not require changes relative to the use of non-porous SOD in order to make process integration a success.

The great majority of the ultra-Low K work reported to date is based on spin-on porous films. As a result, the Symposium attendees anticipated a presentation on CVD ultra-Low K with great interest. Dr. John Macneil from Trikon presented on their Orion™ ultra Low k film ( $K < 2.2$ ). Like most current CVD Low K films, the composition is that of a "carbon-doped oxide" (CDO), alternately called an "organosilicate glass" (OSG), or a "SiCOH" film.

The Trikon CVD ultra-Low K films are similar to spin-on porous SiCOH films in that the mechanical properties are greatly reduced compared to non-porous films; on the positive side, the films appear to have quite small, uniform pores (1-4nm), and low surface roughness. The Orion™ film appears to exhibit unusually high thermal stability compared to other CDO's; Dr. Macneil presented data demonstrating thermal stability of the film up to 500C. These studies focused on the stability of film thickness, stress, FTIR spectroscopy, and dielectric constant measurements after high temperature exposures. Overall, the initial process integration data provided suggests the potential viability of Trikon's CVD ultra-Low K film in future interconnect structures.

The feedback from the attendees at the Innovations in Interconnect Session was almost uniformly positive in their response to both the Session and the individual presentations. With the increasing importance of Advanced Interconnect in semiconductor manufacturing, we can anticipate important future Technical Sessions at SEMICON West featuring important, relevant discussions on a wide range of copper/damascene-related topics.

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<http://dom.semi.org/web/wsemi.nsf/webdocs/E639C63A20A3A4EB88256A9C005E705>  
F 5/12/04

Exhibit C

# Chemical Mechanical Polishing of Polymer Films

DAN TOWERY and MICHAEL A. FURY

AlliedSignal Inc., Advanced Microelectronic Materials, 1349 Moffett Park Drive, Sunnyvale, CA 94089

Strategies to reduce capacitance effects associated with shrinking integrated circuit (IC) design rules include incorporating low resistivity metals and insulators with low dielectric values, or "low- $\kappa$ " materials. Using such materials in current IC fabrication schemes necessitates the development of reliable chemical mechanical polishing (CMP) processes and process consumables tailored for them. Here we present results of CMP experiments performed on FLARE™ 2.0 using a specialized zirconium oxide ( $\text{ZrO}_2$ ) polishing slurry. FLARE™ 2.0 is a poly(arylene) ether from AlliedSignal, Inc. with a nominal dielectric constant of 2.8. In addition, we provide insight into possible removal mechanisms during the CMP of organic polymers by examining the performance of numerous abrasive slurries. Although specific to a limited number of polymers, the authors suggest that the information presented in this paper is relevant to the CMP performance of many polymer dielectric materials.

**Key words:** Chemical-mechanical polishing (CMP), FLARE™ 2.0, low- $\kappa$  dielectric

## INTRODUCTION

Precision polishing processes for scientific applications were described as early as 1695 when Sir Isaac Newton reported using a hand-held pitch lap, tin oxide abrasive, and moisture from his breath, to polish a copper telescope mirror.<sup>1</sup> Since then, polishing science has progressed to include many precision applications, such as ring laser gyro fabrication where surface roughness values below 1.0 Å rms are required, and silicon wafer polishing where surface smoothness and sub-micron thickness uniformity are critical. Technical applications of this nature inspired the development of chemical mechanical polishing (CMP) for integrated circuit (IC) fabrication at IBM in the early 1980s.<sup>2</sup> The prevalence of glass polishing in the optical industry, and the use of  $\text{SiO}_2$ -based materials for inter-metal dielectric (IMD) layers, resulted in extensive contributions to the literature regarding the polishing characteristics of these materials. Mechanistic models describing the chemical and mechanical nature of silica polishing are reasonably well developed<sup>3-6</sup> and have been verified experimentally to some extent. In contrast, only a few citations discuss the polishing of polymeric materials. The polishing of "plastics" is briefly discussed in literature originating

from the precision optical fabrication and ophthalmic industries.<sup>7,8</sup> In these applications, many polymeric materials, such as acrylics, methylmethacrylate, and polycarbonate, are polished with a variety of abrasive slurries derived from  $\text{Al}_2\text{O}_3$ ,  $\text{CeO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{SnO}_2$ ,  $\text{ZrO}_2$ , and mixtures thereof.

In this paper, we present results of CMP experiments performed on FLARE™ 2.0 using a specialized zirconium oxide ( $\text{ZrO}_2$ ) polishing slurry. FLARE™ 2.0 is a poly(arylene) ether from AlliedSignal, Inc. with a nominal dielectric constant of 2.8. FLARE™ 2.0 is an poly(arylene) ether spin-on polymer formulated for use as a stand alone, low  $\kappa$  interlayer dielectric. The high glass transition temperature ( $\geq 400^\circ\text{C}$ ) and low out-gassing characteristics of FLARE™ 2.0 were designed for compatibility with current BEOL processing temperatures. FLARE™ 2.0 has the gap-fill properties for subtractive aluminum etch processing, as well as the one-coat thickness and adhesive properties required for damascene processing. The thermal and chemical stability of FLARE™ 2.0, as well as other poly(arylene) ether materials, make them leading candidates for IC design schemes requiring robust low- $\kappa$  dielectric materials.

## EXPERIMENTAL SET-UP

Blanket FLARE™ 2.0 films were prepared by spin-coating 10KÅ of liquid FLARE™ 2.0 onto prime sili-

(Received April 1, 1998; accepted June 2, 1998)



FOR IMMEDIATE RELEASE

Exhibit D

## NOVELLUS INTRODUCES INTEGRATED LOW-K SOLUTION FOR NEXT-GENERATION COPPER CHIPS

San Jose, California -- Jun. 11, 1999 -- Novellus Systems Inc. (Nasdaq:NVLS), the productivity and innovation leader in thin film deposition technologies for the global semiconductor industry, today unveiled its new CORAL<sup>®</sup> family of production-worthy low dielectric constant (low-k) films for advanced devices. Designed for deposition on Novellus' enhanced SEQUEL Express<sup>™</sup> platform--also introduced today--the CORAL family of films completes the company's offerings of dielectric films required to build copper dual damascene structures extendable down to sub-0.1-micron technologies. A 300 mm version of the CORAL system will be available for shipment later this year, running on the same production-proven platform.

Low-k dielectrics and copper interconnects are two key elements necessary to build the advanced integrated circuits (ICs) required for today's increasingly powerful consumer electronics. These applications require faster and smaller ICs that have increased functionality, yet consume less power. To build these chips it is necessary to lower both the effective interconnect resistivity and capacitance. Copper addresses the low-resistivity side of the equation. The proper combination of low-k via and line dielectrics and thin films for copper barriers and etch stops solves the low-capacitance side.

"A year ago we enabled the development of copper interconnects for the semiconductor industry with the introduction of SABRE<sup>™</sup>, the first production-worthy copper electrofill tool, and our INOVA<sup>™</sup> tool, which provides a superior deposition solution for the required barrier and seed layers. Today, with the introduction of our CORAL-based, low-k dielectric solution, which is completely optimized for copper-based devices, Novellus has extended the 'Road to Damascus' further into the future," said Novellus Chairman and Chief Executive Officer Richard S. Hill. "The combination of these low-k and via and line dielectrics and thin film barrier technologies will enable the manufacture of advanced devices down to the sub-0.1-micron node, enabling the development and production of powerful electronic systems designed to meet consumer demands."

Semiconductor manufacturers require production-worthy low-k dielectric films that are extendible and have a low cost of ownership (CoO). Novellus has leveraged its historical expertise in blanket dielectrics and developed the CORAL family of carbon doped oxide films, with dielectric constants ranging from 3.3 to less than 2.5. The films are easily integrated into the dual damascene process flow with their dielectric etch, metal deposition and chemical mechanical polishing (CMP) processes, developed in collaboration with Novellus' Damascus Alliance partners, at a cost comparable to traditional dielectric films and other competitive offerings.

Recognizing that barrier layers are critical to developing a truly low-k integrated stack, Novellus has developed a variety of dielectric barriers and etch stops for low-k films. "By optimizing CORAL with the right thin films, we are able to deliver the full benefits of dielectric constant reduction," said Novellus Vice President for Dielectrics Wilbert van den Hoek. "For instance, a CORAL film with a dielectric constant of 2.7, optimized with an oxide-based barrier with a



CORAL film with a dielectric constant of 2.7, optimized with an oxide-based barrier with a dielectric constant of 4.1, yields an effective capacitance reduction of up to 40 percent."

According to Novellus' research, the market for damascene dielectric films is expected to grow to more than \$500 million by 2003. With the introduction of the CORAL family of films, Novellus continues to extend its tradition of offering the lowest risk, highest performance and lowest cost thin film deposition solutions for the most advanced semiconductor process technologies.

Designed for CORAL and Novellus' other dual damascene barrier films, the SEQUEL Express deposition system leverages the production-proven multi-station sequential deposition architecture to deliver superior reproducibility. The SEQUEL Express system has a maximum throughput in excess of 110 wafers per hour (wph) for thin films and in excess of 80 wph for CORAL films. It delivers up to 40 percent higher capital productivity, up to 100 percent higher productivity per square foot of footprint and up to 40 percent lower CoO than competing CVD systems.

Novellus will begin volume shipments of its CORAL low-k dielectric process on SEQUEL Express later this year and has received orders from several customers for CORAL film upgrade kits. The company has already shipped approximately 20 SEQUEL Express systems for the deposition of other dielectric films required for dual damascene to customers worldwide.

Novellus will feature the CORAL family of films and SEQUEL Express, along with its *Damascus® Complete Copper™* line of products and services at the Yerba Buena Center, adjacent to the Moscone Center, San Francisco, during SEMICON West '99, to be held July 12-14.

#### About Novellus Systems

Novellus Systems Inc. manufactures, markets and services advanced automated wafer fabrication systems for the deposition of thin films. Novellus deposition systems are designed for high-volume production of advanced, leading-edge semiconductors at the lowest overall cost. Headquartered in San Jose, Calif., with subsidiaries in the United Kingdom, France, Germany, the Netherlands, China, Japan, Korea, Singapore and Taiwan, Novellus is a publicly traded company on the Nasdaq stock exchange (Nasdaq:NVLS). Additional information about the company is available on Novellus' home page on the World Wide Web, located at <http://www.novellus.com>.

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Exhibit E

The screenshot shows the Applied Materials website interface. At the top left is the Applied Materials logo. A navigation bar contains links for NEWS, PRODUCTS, INVESTORS, CAREERS, and ABOUT. A search bar is located on the right. On the left side, there is a sidebar menu with sections: Products (listing Silicon Products, Factory Efficiency Products, Flat Panel Display Products, Mask Products, Customer Training, Technical Glossary, and Contact Information), Related Links (listing Product Features and Press Releases), and Contact Us (listing Keep Me Informed, Legal Information, and Privacy Statement). The main content area is titled "Applied Producer Black Diamond" and contains two paragraphs of text. To the right of the text is a vertical list of links: Back to D Deposition, CVD Low, Applied BLOk, Applied Black T, HDP-CVD, PECVD, SACVD, and About CVI. The footer at the bottom left contains copyright information for 2004.

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## Applied Producer Black Diamond

Black Diamond is a silicon oxide-based CVD low k film that provides a dielectric constant of 3.0. This film is designed for copper damascene interconnects and enables Applied Materials' customers to achieve superior performance for sub-130nm device technology.

This unique CVD low k film provides sufficient hardness and relatively straightforward integration, exhibiting excellent resistance to moisture, as well as a stable k-value after etching and photoresist removal. In addition to allowing direct patterning without the use of capping layers and good CD (critical dimension) control, Black Diamond demonstrates excellent cracking resistance and good thermal conductivity, enabling full trench/via and multi-level metal integration. The film also provides sufficient hardness for direct polishing without the use of hardmasks or capping layers.

Black Diamond is available on the Producer platform. Use of conventional CVD hardware enables extension of existing equipment to multiple low k applications and generations and avoids the use of costly proprietary chemicals.

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## Z3MS™

### Trimethylsilane

#### Product Description

Dow Corning® Z3MS™ CVD Precursor is a silicon source specialty gas engineered for PECVD processes used to deposit thin film dielectrics.

#### Air Products: Global Supplier of CVD Low-k Dielectric Precursors

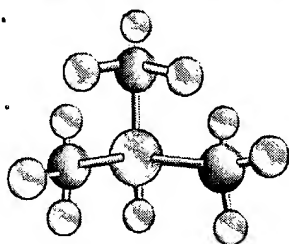
Air Products offers a comprehensive low-k package, including a variety of high-purity electronic specialty gases, its Schumacher unit's low-k chemical product line, delivery equipment and MEGASYS® on-site management services to maximize productivity and speed the development of new systems.

The company's portfolio of interlayer dielectric (ILD) precursor products includes SiH<sub>4</sub>, 1MS, SiF<sub>4</sub> and chlorosilanes (TCS, STC, DCS).

Air Products' Schumacher unit offers several proven ILD products, including TEOS, ZTOMCATS™, Z4MS™ and Z2DM™ CVD low-k precursors, and our MesoELK™ SiO<sub>2</sub> precursor.

Air Products has substantially increased its offering for fluorinated silicon glass by significantly ramping up SiF<sub>4</sub> capacity in Morrisville, Pennsylvania and in Shihwa, Korea.

In addition, Air Products offers a full line of gas and chemical delivery equipment providing a high degree of safety and effective use of its low-k products. Offerings include the company's ChemGuard® and GASGUARD® gas cabinets.



Trimethylsilane (Z3MS)  
Molecule

Table 1

#### Physical Characteristics:

Formula	(CH <sub>3</sub> ) <sub>3</sub> SiH
Molecular Weight (g/mol)	74.19
Approx Gas Density (mg/cc)	3
Vapor Pressure at 25°C	1400 Torr

Table 2

#### Product Specification

Total Purity	≥99.995 vol%
Carbon Dioxide	≤2.0ppmv
Carbon Monoxide	≤2.0ppmv
Nitrogen	≤2.0ppmv
Oxygen and Argon	≤2.0ppmv
Total Chlorides	≤1.0ppmv
Total Hydrocarbons	≤5.0ppmv
Water	≤1.0ppmv
Aluminum	≤5.0ppbw
Arsenic	≤5.0ppbw
Barium	≤5.0ppbw
Bismuth	≤5.0ppbw
Calcium	≤5.0ppbw
Cobalt	≤5.0ppbw
Chromium	≤5.0ppbw
Copper	≤5.0ppbw
Gallium	≤5.0ppbw
Indium	≤5.0ppbw
Iron	≤5.0ppbw
Lead	≤5.0ppbw
Lithium	≤5.0ppbw
Magnesium	≤5.0ppbw
Manganese	≤5.0ppbw
Nickel	≤5.0ppbw
Potassium	≤5.0ppbw
Sodium	≤5.0ppbw
Strontium	≤5.0ppbw
Thorium	≤5.0ppbw
Tin	≤5.0ppbw
Titanium	≤5.0ppbw
Tungsten	≤5.0ppbw
Uranium	≤5.0ppbw
Zinc	≤5.0ppbw

***For More Information***

This datasheet has provided only a brief overview of Air Products' wide range of low-k solutions. If you would like more information, please contact:

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Exhibit G

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Chen, W. (Dow Corning / MI) Deis, T. A. (Dow Corning / MI) Moyer, E. S. (Dow Corning / MI) Lee, Hae-Jong. Lin, E. K. Wang, H. Wu, W. L. (POLYMERS DIVISION - 854)

#### **Structural Comparison of Hydrogen Silsesquioxane Based Porous Low-k Thin Films Prepared With Varying Process Conditions** *Chemistry of Materials* - April 01, 2002

The structure of hydrogen silsesquioxane (HSQ) based porous low dielectric constant (low-k) films (XLK™) prepared with varying process conditions are characterized using a combination of high energy ion scattering, x-ray reflectivity (SXR), and small angle neutron scattering (SANS). We measure the film thickness, average mass density, density depth profile, wall density, porosity, average pore size, pore spacing, pore connectivity, and atomic composition. We compare samples with varying dielectric constants and degrees of cure or Si-H bonding fraction. The structural parameters are correlated with the chemical bond structure as measured by Fourier transform infrared (FTIR) spectroscopy. The density profiles of the porous films were uniform with a slight densification observed at the film surface. Films with similar k values but varying degrees of cure have almost identical structural characteristics. Lower dielectric constant films have larger porosities and average pore sizes, but lower wall densities. The process conditions used to alter the dielectric constant affect not only the porosity, but also many other structural parameters such as the wall density.

*Keywords: porous low-k dielectric , small angle neutron scattering (SANS) , density profile , wall density , hydrogen silsesquioxane , x-ray reflectivity , density profile , porosity , wall density*

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